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PATENT SPECIFICATION

Inventors: IMPERIAL CHEMICAL INDUSTRIES LIMITED.

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ERRATUM

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In the heading on Page 1, delete "Inventors: IMPERIAL CHEMICAL INDUSTRIES LIMITED."

THE PATENT OFFICE,
10th July, 1950.

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condition. or substances that tend to form more or less consistent masses, the division of which requires additional operations.

15 It is known that when some substances as chlorinated rubber, are separated from their solution by the addition of water or another liquid not miscible with the solvent, there is separated a mass of spongy appearance which is not suited for further manipulations or treatment unless it is previously divided into small pieces. This division is usually carried out after centrifuging or drying the mass and it is a complicated and costly operation.

25 This invention has for its object to carry out the precipitation in such manner that the material separated is obtained directly in a divided form, as in filament, clot, flakes or powder form.

30 The process according to this invention consists in subjecting a thin stream or sheet of the solution to the action of thin jets of a liquid that does not dissolve the substance to be precipitated and is not miscible with its solvent, whereby the stream or sheet of solution is divided into fine drops which let go, each separately, the precipitable sub-

Where an auxiliary liquid is used that has a considerably higher boiling point than the solvent, precipitation is promoted if the auxiliary liquid is previously heated so as to accelerate evaporation of the solvent. The vapors of the solvent are recovered, while all or part of the auxiliary liquid carries away the particles of the precipitate formed, which are insoluble therein and show no tendency to agglomerate and adhere into a mass.

60 In carrying out the invention, an emulsifying agent may be added to the auxiliary liquid in order to form an emulsion of the solution in the auxiliary liquid and obtain the precipitate from such emulsion.

75 The accompanying drawing illustrates diagrammatically by way of example an apparatus adapted for carrying out the above process.

This apparatus comprises a chamber or column A at the top of which opens a pipe with a vertical nozzle B for delivering the solution. Under the nozzle B open a plurality of converging nozzles C for supplying under pressure the auxiliary liquid at elevated temperature

which is used for precipitation. A ring of jets D adjacent the walls of the chamber A is also fed with auxiliary liquid which is thus caused to trickle down the walls.

The stream of solution issuing from the nozzle B is broken by the thin jets of liquid supplied under pressure by the nozzles C and is divided into drops. Where an auxiliary liquid is used that has a considerably higher boiling point than the solvent and is introduced at a higher temperature than the latter, the heat of the former causes evaporation of the solvent in the drops of solution as they are distributed in the chamber A, so that the precipitate separates in said chamber in a finely divided state. The vapors of the solvent which may be mixed with vapors of the auxiliary liquid, leave the chamber A at G, while the precipitate is carried by the liquid trickling from the ring D down to the bottom of the chamber.

The suspension of precipitate in the auxiliary liquid leaves the chamber A and enters a separator E from which the precipitate is withdrawn, while the liquid is sucked by a pump F and led back to the chamber A in part through the nozzles C and in part through the ring D.

The heat spent for vaporising the solvent and the part of the auxiliary liquid which is carried away from the chamber A in the form of vapor, are compensated by introducing boiling liquid or vapor at H, preferably in the liquid at the bottom of the chamber.

As a first example of carrying out the invention, we may mention the precipitation of chlorinated rubber in solution in carbon tetrachloride. The syrupy mass fed through the nozzle B is subjected to the action of jets of water at a temperature of 90 to 95° C. supplied through the nozzles C. Filaments of chlorinated rubber are seen to be torn from the central stream by the action of the jets. The vapors of carbon tetrachloride mixed with water vapor leave the apparatus and are separated by cooling. Heated water vapor, on the other hand, is introduced at H into the chamber A.

As another example, we shall mention the precipitation of benzene hexachloride from its solution in benzene. Here again the auxiliary liquid is water, that may be mixed with an emulsifying agent, and the product is obtained in form of a fine powder.

These two examples do not limit the invention, as the process is applicable generally to solutions that relinquish the dissolved substances, as a result either

of vaporisation of the solvent, provided the auxiliary liquid shall not be miscible with the solvent, neither should it be capable of dissolving the precipitate. The addition of an emulsifying agent to the auxiliary liquid is favorable to the production of a precipitate of great fineness.

It is obvious that the form of the apparatus described may be varied. Thus, for example, the outlet for the precipitate may be arranged axially at the bottom of the chamber A, and separated from the liquid by centrifuging, the liquid inlet may extend in any other direction, the precipitation chamber may be heated outwardly, and the apparatus may be provided with temperature exchangers and other auxiliary devices.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the precipitation of substances from solutions containing same by adding thereto an auxiliary liquid which is neither a solvent of said substance nor miscible with its solvent, characterised by the fact that a thin stream or sheet of the solution is subjected to the action of thin jets of said auxiliary liquid, whereby the stream or sheet of solution is broken up into fine drops which let go, each separately, the precipitable substance contained therein and thus supply the precipitate in a state of division.

2. A process according to claim 1, characterised by the fact that an auxiliary liquid is used that has considerably higher boiling point than the solvent and is heated prior to its introduction into the solution so as to cause the solvent to vaporise, the mixture of vapours of the solvent and of the auxiliary liquid being separated in the usual manner in order to recover the solvent.

3. A process according to claim 1 or 2, characterised by the fact that the auxiliary liquid is delivered partly through nozzles directed towards the stream or sheet of solution, and partly through openings arranged along the inner walls of the precipitation chamber.

4. A process according to claim 1 or 2, characterised by the fact that an emulsifying agent is added to the auxiliary liquid.

5. An apparatus for carrying out the process according to claims 1 and 3, comprising a precipitation chamber, means for delivering the solution in form of a thin sheet or stream, a set of auxiliary liquid delivery nozzles converging towards said sheet or stream a row of

[This Drawing is a reproduction of the Original on a reduced scale.]

